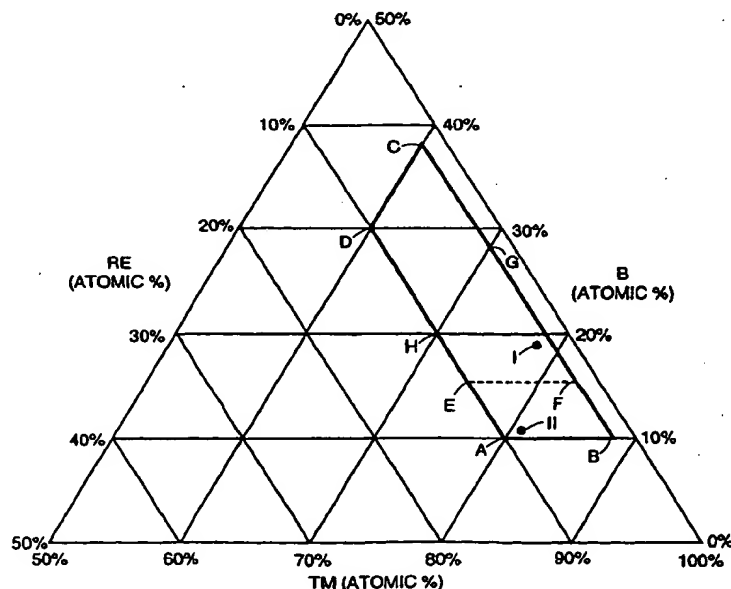


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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US00/05657 <b>(22) International Filing Date:</b> 3 March 2000 (03.03.00) <b>(30) Priority Data:</b> 09/263,347 5 March 1999 (05.03.99) US <b>(71) Applicant:</b> PIONEER METALS AND TECHNOLOGY, INC. [US/US]; 60 State Street, Boston, MA 02109 (US). <b>(72) Inventor:</b> KEEM, John, E.; 1641 Lone Pine Road, Bloomfield Hills, MI 48302-2642 (US). <b>(74) Agents:</b> LAMPERT, James, B. et al.; Hale and Dorr LLP, 60 State Street, Boston, MA 02109 (US).		<b>(81) Designated States:</b> European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>

**(54) Title:** MAGNETIC MATERIAL**(57) Abstract**

Magnetic materials having a coercivity not less than about 1000 Oersted are prepared in a single step procedure. A molten mixture of a desired composition having a relatively high boron content is cooled at a rate slower than about  $10^5$  degrees Celsius per second. Preferably, the molten mixture is cooled by depositing it on a chilled surface such that it forms a layer between about 120 and about 300, and preferably between about 120 and about 150, microns thick.

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## MAGNETIC MATERIAL

### Field of the Invention

This invention relates to permanent magnetic materials of RE-TM-B alloys, and, more particularly, to methods of producing alloyed Nd/Pr-Fe-B alloyed powders.

### Background of the Invention

This invention relates to magnetic materials comprising one or more transition metals, one or more rare earth elements and iron. A variety of such materials are known. See, for example, US Patents Nos. 4,374,665, 4,402,770, 4,409,043 and 4,533,408 of Koon which disclose magnetic alloys comprising iron, lanthanum and a lanthanide, and boron; US Patents 4,802,931 and 4,851,058 to Croat which disclose single phase TM-RE-B alloys, US Patent No. 4,935,074 of DeMooij et al. which discloses a material including iron, a relatively high percentage of boron, and Nd and/or Pr; US Patent 5,071,493 to Mizogochi et al., and US Patent 4,770,723 to Sgawa et al. Methods of preparing such magnetic materials are taught in U.S. Patent Nos. 4,867,785 and 5,116,434 to Keem et al., and US Patents Nos. 4,715,891 and 4,753,675 to Ovshinsky et al.

In general, the magnetic material is prepared in two-steps. The first step is to prepare a substantially homogeneous master alloy of the desired composition of the various components, and then to break the alloy into relatively large, e.g., 1 cm pieces, that can be easily remelted for later processing. The second step is to remelt the alloy in a crucible, and then to quench and rapidly solidify (e.g., 25-30 micron and not over 50 microns thick) homogeneous ribbon. Typically the quenching and rapid solidification is

accomplished by passing the melted material through a small (e.g., 1 mm in diameter) orifice onto a moving chill surface, e.g., a rotating chill surface such as that shown in aforementioned U.S. Patent No. 4,867,785.

Typically, the ribbon is then crushed into fine powder, the powder is then heat treated and magnetically separated (e.g., as disclosed in U.S. Patent No. 5,116,434) to separate powder having high magnetic parameters from that having lower magnetic parameters, and the high magnetic parameter powder is made into permanent magnets.

The above described procedure, and particularly the quenching and rapid solidification, is expensive and subject to a number of potential problems and difficulties. For example, unsatisfactory product may result from variations in the flow from the crucible caused by alloy/crucible reaction products, slag or products of reaction between the atmosphere and the molten alloy at the crucible orifice that can clog or impede flow through the small crucible orifice, or for relatively small variations in the chill surface temperature or speed that affect the quench rate and/or ribbon thickness.

There has been, and remains, a need for a process that is less expensive and has fewer difficult-to-control parameters.

#### Summary of the Invention

According to the present invention, a class of alloyed magnetic materials, i.e., materials having a relatively high boron content, are prepared in a single step procedure. A molten mixture of the desired composition is cooled at a rate slower than about  $10^5$  degrees Celsius per second, and preferably more than about  $10^4$  degrees Celsius per second. Without the need for conventional

rapid quenching, a powder of the slow-cooled material may then be heat treated, and, optionally, magnetically separated. Surprisingly, such slow cooled material has been found to have magnetic properties superior to those of material of identical composition made in the conventional two-step, rapid quenching procedure.

In some preferred practices of the invention, the molten mixture is solidified by depositing it on a heat conducting surface such that it forms a layer between about 120 and about 300, and preferably between about 120 and about 150, microns thick, and the relatively thick layer of slow-cooled material is then crushed into powder before heat treating. In these and other preferred practices of the invention, the molten mixture contains more than about 10 (and most preferably not less than about 20) atomic percent boron and not more than about 10 atomic percent of a rare earth. Preferably at least 90 percent by weight of the rare earth content is Nd, Pr or a mixture thereof and any other rare earth constitute less than 10 percent of the total rare earth content. In most preferred practices, the molten mixture includes at least 60 atomic percent, and preferably at least 70 atomic percent, Fe.

The above, and other objects, features and advantages of the invention will appear from the following detailed description, taken in conjunction with the attached drawings in which:

Figure 1 is a partial tertiary phase diagram illustrating the compositional make-up of the permanent magnet material of the present invention; and

Figure 2 is a flow chart schematically illustrating the procedure of the present invention.

### Detailed Description

Permanent magnet materials made according to the present invention include (a) one or more transition metals, (b) one or more rare earth metals, and (c) boron. They may also include relatively small amounts of one or more auxiliary glass formers such as silicon or aluminum.

Boron comprises between about 10 atomic percent and about 30 atomic percent, and preferably not less than about 20 atomic percent, of the material. The rare earth (RE) component is neodymium, praseodymium or a mixture of the two, preferably neodymium, and comprises between about 3 atomic percent and about 10 atomic percent of the material; up to about 10 percent of the total rare earth content of the alloy may be other rare earths. The transition metal (TM) typically is iron, cobalt, or a mixture thereof, preferably iron, and may include relatively minor amounts of one or more of titanium, vanadium, zirconium, niobium, molybdenum, hafnium, chromium or nickel. Except for minor amounts of a glass former such as silicon or aluminum, the transition metal component comprises the balance, not less than about 60 atomic percent, and preferably not less than about 70 atomic percent, of the material.

The relative amounts of the various components are interdependent, as shown in the partial ternary phase diagram of Figure 1. Specifically, the points designated as A, B, C and D in Figure 1 define a region of the phase diagram, in the general shape of a parallelopiped, the boundaries of which define the compositional range of permanent magnet materials made according to the present invention. It will be noted that the materials have a minimum boron content of about 10 atomic percent, and a maximum RE content of about 10 atomic percent. The preferred compositional range is the generally trapezoidal

region defined by the points designated as E, F, G and H. Materials within this preferred compositional range have a minimum boron content of about 15 atomic percent and a minimum TM content of about 70 atomic percent.

As shown in Figure 2, the first step in producing the magnetic material according to the present invention is to place the TM, RE and B components of the material in a ceramic-lined crucible which is in turn placed in a vacuum-induction furnace. (Step 200) The crucible and its contents are heated to form a homogenous alloy melt. (Step 210) As in conventional rapid solidification procedures, the melted alloy is then solidified. (Step 220) Conventionally, the material is rapidly solidified by depositing the melt onto a cooling surface at a fairly slow mass flow rate (e.g., by directing the material onto the surface through a funnel having an orifice not more than about 1 mm orifice in diameter) so that it cools at a rate of not less than about  $10^6$  degrees Celsius per second. In the practice of the present invention, the melt is cooled at a much slower rate than in conventional rapid solidification, i.e., at a rate slower than about  $10^5$  degrees Celsius per second and preferably faster than about  $10^4$  degrees Celsius per second. This slower solidification rate can be achieved using a number of solidification procedures. For example, it is conventional to rapidly cool a liquid homogeneous melt using rotating cup or other forms of gas atomization. The parameters that control the cooling rate in these atomization procedures are understood by those skilled in the art and, in the practice of the present invention, these procedures can readily be modified so that the atomized particles from the liquid homogeneous melt cool at a rate in the about  $10^4$  to about  $10^5$  degrees Celsius per second range discussed above, rather than at the faster conventional rapid quench rate.

In the preferred practice of the present invention, the relatively slower cooling rate is achieved by adjusting the mass flow rate at which the liquid melt impinges onto the wheel or other cooling surface. Typically, a much greater mass flow rate, e.g., about 4 kg per minute, as compared to the less than 0.5 kg per minute mass flow rate typical in conventional rapid solidification procedures, is achieved by directing the molten material from the melt onto the cooling surface through a larger orifice that is much larger, e.g., is about 3 mm in diameter, and thus has a cross-sectional area about an order of magnitude larger, than that used in conventional rapid solidification. Largely because of the greater mass flow rate, the melt cools at a much slower rate than when rapidly quenched, and solidifies to form a layer that is significantly thicker (e.g., between about 120 and 200 microns, and preferably about 120 to 150 microns, thick) than the ribbon (typically 25-35 microns thick) produced during conventional rapid solidification. In this respect, it will be recognized that the thickness of the deposited layer is directly related to the cooling rate (i.e., the faster the cooling rate the thinner the layer), and that those skilled in the field are able quite accurately estimate the cooling rate on the basis of the deposited thickness.

The thus produced layer is then crushed to form a fine powder (Step 230); however, it will be recognized that this step may be omitted when the solidification process itself, e.g., an atomization procedure, produces a sufficiently fine powder. However produced directly or by crushing, the powder is heat-treated in a rotary kiln in the usual manner (Step 240), e.g., at a temperature in the range of about 600 to about 750, and preferably about 680, degrees Celsius. After heat treating, the powder has a coercivity of more than



1000 Oersteds. Optionally, the powder is magnetically separated (Step 240) before forming into a permanent magnet (Step 250).

Powders having the compositions discussed above and produced using the procedure of the present invention have been found to have coercivity and other magnetic properties equal or superior to those of powders having the same composition made according to conventional two-step, rapid quenching processes. However, it has also been found that both the cooling rate and the composition of the material are important; powders having compositions outside of the range discussed above, e.g., powders made by a relatively slow-cooling process have properties that are significantly poorer than powders of the same composition made according to conventional two-step, rapid quenching processes if the boron content of the material is too low, i.e., is less than about ten atomic percent, or rare earths other than Nd or Pr constitute more than about ten atomic percent of the total rare earth content.

This is illustrated by the following Table that compares a magnetic material of the present invention to four other magnetic materials. The initial entries in the Table identify the composition of the various materials. The composition of the material of the present invention (Example 5) and those of Examples 3 and 4 are approximately the same; and the composition of the material of Example 1 has about the same B, TM and overall RE content. The compositions of all four of these materials are substantially that identified as point I on Figure 1. Similarly, the somewhat different composition of the material of Example 2 is identified as point II on Figure 1. Other entries in the Table refer to variations in the processing procedure: e.g., "Orifice" is the diameter, in mm, of the orifice through which the melt flows onto the cooling

surface; "Thickness" and "Width" are the width and thickness of the ribbon or layer formed on the cooling surface; "Mass Flow" is the rate at which the melt flows onto the cooling surface; and "Wheel Speed" is the speed of the circumference of the circular cooling surface. In all of the examples, the materials were subjected to the same "Heat Treatment", i.e., 680 degrees C. in a rotating kiln. "BHmax", "Br" and "Hci" are magnetic properties of the powder produced from the various materials.

Composition (at%)	Example 1	Example 2	Example 3	Example 4	Present Invention
Nd	3.8	8.5	3.98	4.05	4.0
La	0.56	-	-	-	-
B	18.7	10.5	18.38	19.02	19.0
Fe	Balance	Balance	Balance	Balance	Balance
Orifice (mm)	3	3	3	1	3
Thickness (mm)	150	120	200-300	30	120
Width (mm)	8-12	8-12	6-10	2-3	8-12
Mass flow (kg/min)	4	5	5	0.4	4
Wheel speed (m/sec)	12	12	12	25	12
Heat treatment (C)	680	680	680	680	680
BH max (MGOe)	<8	<5	<8	11	13
Br (kG)	<9	<7	9	10	11
Hci (kOe)	<2	<6	1.2	2.6	3.4

The material of Example 4 was produced using conventional procedures, i.e., a master alloy was formed in a first step, and the alloyed material was then rapidly solidified in the conventional manner in a second step. All of the other materials, i.e., those of Examples 1 through 3 and 5, were produced by

depositing the melted material onto a cooling surface in such a way that it cooled more slowly, i.e., at a rate slower than about  $10^5$  degrees Celsius per second but more than about  $10^4$  degrees Celsius per second, and formed a thicker layer, than in conventional rapid quenching.

From the Table, it will be noted that materials having compositions of about 4 atomic percent Nd, about 18-19 atomic percent Boron, and balance FE, had good properties when produced using conventional rapid solidification techniques (Example 4), and had significantly better magnetic properties when produced using the procedure of the present invention (Example 5). However, a material of substantially the same composition (Example 3) had unsatisfactory properties if the melted material was cooled too slowly, e.g., when deposited on the cooling surface in such a way that the too-slow cooling rate resulted in a deposited layer more than 200-300 microns thick (Example 3).

It will similarly be noted from the Table, that, even when produced using a procedure in which the material was cooled at the rate (more than  $10^4$  but less than  $10^5$  degrees Celsius per second) desired in the practice of the present invention, powder materials that included too little boron (e.g., Example 2) or which had about the same overall B, RE and TM content as materials of the present invention but in which lanthanum was more than 10 percent of the total rare earth content (e.g., Example 1), were unsatisfactory.

It will be appreciated that the particular examples shown in the Table are exemplary only and do not limit the scope of the present invention. Rather, the present invention, and its advantages, encompass materials of other compositions within the scope of the claims when produced using the claimed

procedures. Thus, other embodiments of the invention will be within the scope of the following claims.

What is claimed is:

1. The method of producing a magnetic material having a coercivity greater than 1000 Oersted comprising the steps of:  
providing a molten mixture including boron, one or more rare earths and one or more transition metals; and  
cooling said mixture at a rate slower than about  $10^5$  degrees Celsius per second, the molten mixture containing not less than about ten atomic percent boron, not less than about 60 atomic percent transition metals, and not more than about 10 atomic percent rare earths.
2. The method of claim 1 wherein the rate at which said mixture is cooled faster than about  $10^4$  degrees Celsius per second.
3. The method of claim 1 wherein said molten mixture is cooled by impinging said molten mixture on a heat conducting surface so that said mixture forms a layer having a thickness between about 120 and about 300 microns.
4. The method of claim 1 wherein the thickness of said layer is between about 120 and about 150 microns.
5. The method of claim 1 wherein said mixture includes between about 10 and about 30 atomic percent boron.
6. The method of claim 5 wherein said mixture includes between about 15 and about 20 atomic percent boron.

7. The method of claim 1 including the steps of heat treating a powder of said mixture, and thereafter producing a permanent magnet including said powder.
8. The method of claim 7 including the step of cooling said mixture to form a layer thereof and thereafter crushing said layer into said powder.
9. The method of claim 7 including the step of magnetically separating said powder prior to producing said magnet.
10. The method of claim 1 wherein at least one of said rare earths is selected from the group including Nd, Pr, and a mixture thereof.
11. The method of claim 1 wherein at least 90% by weight of said rare earths comprise Nd, Pr, or a mixture thereof.
12. The method of claim 11 wherein not more than 10 percent by weight of said rare earths comprise a rare earth other than Nd or Pr.
13. The method of claim 1 wherein said mixture includes between about 3 and about 10 atomic percent rare earth.
14. The method of claim 1 wherein said molten mixture includes at least 60 atomic percent of one or more transition metals.

15. The method of claim 1 wherein said molten mixture includes at least 70 atomic percent of one or more transition metals.
16. The method of claim 1 wherein said transition materials are selected from the group consisting of titanium, vanadium, zirconium, niobium, hafnium, chromium, nickel and iron.
17. The method of claim 1 wherein the major component by weight of said transition metals is Fe.
18. The method of claim 1 wherein the composition of said molten mixture is within the bounds of the parallelepiped whose corners are defined by points A, B, C and D of Figure 1.
19. The method of claim 19 wherein the composition of said molten mixture is within the bounds of the trapezoid whose corners are defined by points E, F, G and H of Figure 1.
20. The method of producing a magnetic material having a coercivity greater than 1000 Oersted comprising the steps of:  
providing a molten mixture including boron, one or more rare earths and one or more transition metals, the molten mixture having not less than about ten atomic percent boron; and

cooling said mixture by impinging said molten mixture on a heat conducting surface such that said mixture solidifies to form a layer having a thickness between about 120 and about 300 microns.

21. The method of claim 20 wherein said mixture is solidified to form a layer having a thickness between about 120 and about 150 microns.
22. The method of claim 20 wherein the composition of said molten mixture is within the bounds of the parallelepiped whose corners are defined by points A, B, C and D of Figure 1.
23. The method of claim 22 wherein the composition of said molten mixture is within the bounds of the trapezoid whose corners are defined by points E, F, G and H of Figure 1.
24. The method of claim 22 wherein not more than 10 percent by weight of said rare earths comprise a rare earth other than Nd or Pr.
25. The method of claim 22 wherein transition materials are selected from the group consisting of titanium, vanadium, zirconium, niobium, hafnium, chromium, nickel and iron.
26. The method of claim 20 including the steps of producing a powder from said layer, heat treating said powder, and thereafter producing a permanent magnet including said powder.



27. The method of producing a magnetic material having a coercivity greater than 1000 Oersted comprising the steps of:

providing a molten mixture including about 10 to about 30 atomic percent boron, about 3 to about 10 atomic percent one or more rare earths and not less than about 60 atomic percent one or more transition metals; and

cooling said mixture at a faster rate than about  $10^4$  degrees Celsius per second and about slower than about  $10^5$  degrees Celsius per second by impinging said molten mixture on a heat conducting surface such that said mixture solidifies to form a layer having a thickness between about 120 and about 300 microns.

28. The method of claim 25 wherein said mixture is solidified to form a layer having a thickness between about 120 and about 150 microns.

29. The method of claim 25 wherein the composition of said molten mixture is within the bounds of the parallelepiped whose corners are defined by points A, B, C and D of Figure 1.

30. The method of claim 27 wherein the composition of said molten mixture is within the bounds of the trapezoid whose corners are defined by points E, F, G and H of Figure 1.

31. The method of claim 27 wherein not more than 10 percent by weight of said rare earths comprise a rare earth other than Nd or Pr.

32. The method of claim 27 wherein said transition metals are selected from the group consisting of titanium, vanadium, zirconium, niobium, hafnium, chromium, nickel and iron, and the major fraction by weight of said transition metals is iron.

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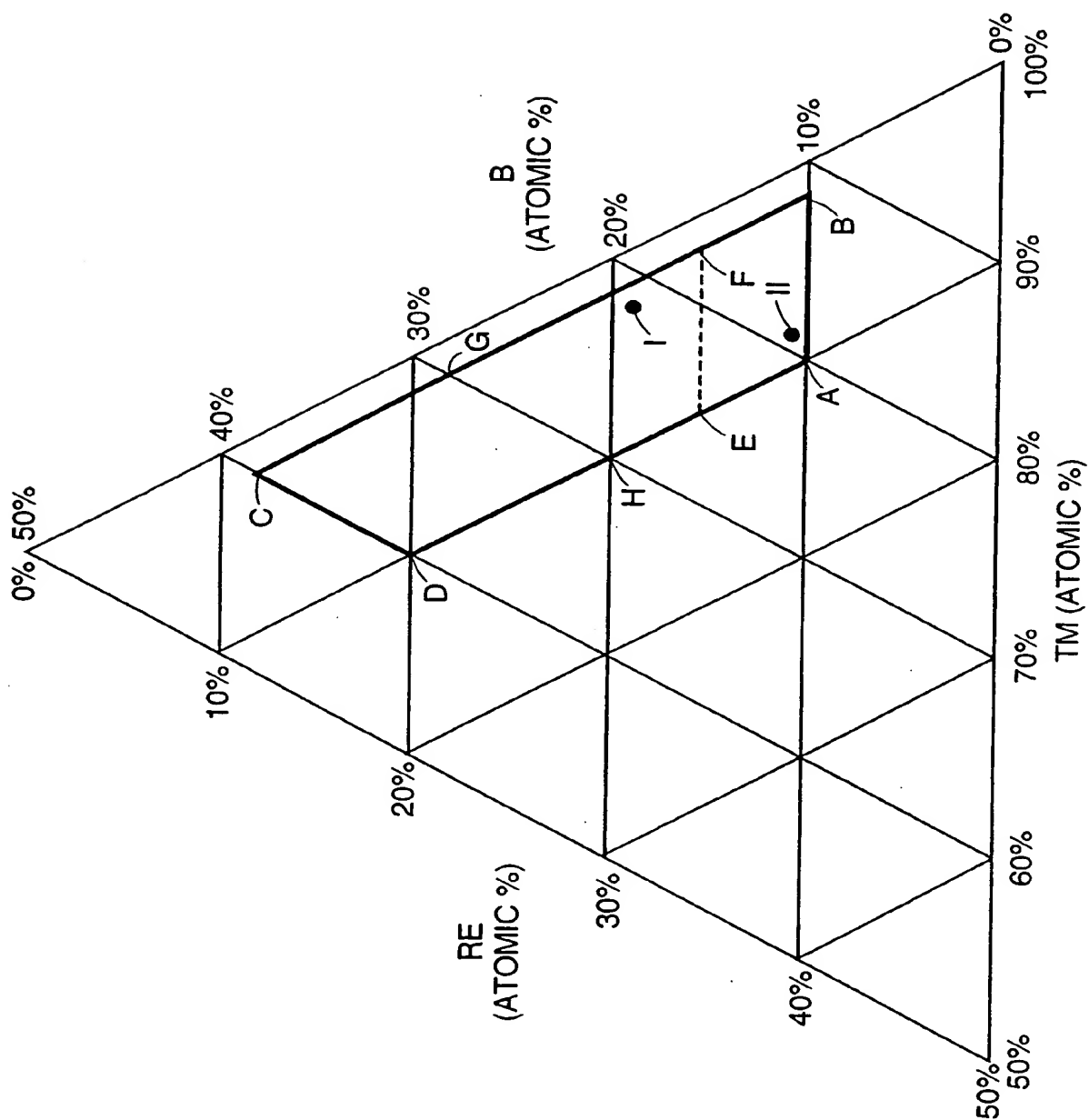


FIG. 1

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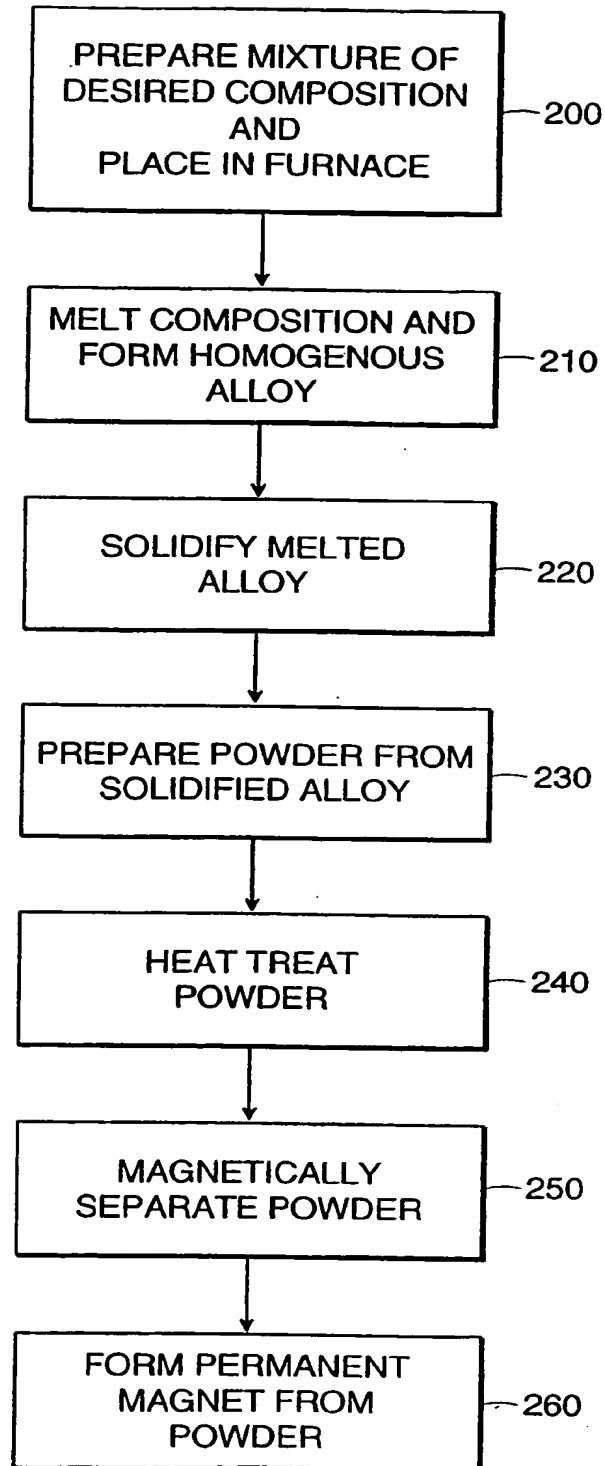


FIG. 2

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 00/05657

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 H01F1/057

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 H01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	EP 0 542 529 A (SUMITOMO SPEC METALS) 19 May 1993 (1993-05-19) page 4, line 9 - line 25 page 5, line 15 - line 21; claims 1,2,4,8,10-12 --- -/-	1,5-8, 10-15,17

☒ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

14 June 2000

Date of mailing of the international search report

23/06/2000

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# INTERNATIONAL SEARCH REPORT

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

In: ational Application No

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